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Automation of Karl Fischer Titration for Animal Pharmaceutical Product Testing at Elanco Animal Health

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Elanco

Company Confidentiality Disclaimer

Much of my work this summer is proprietary information for Elanco Animal Health. The details described are extremely broad and do not contain any data or product names.

Experimental Methods:

Comparability Study: A high, middle, and low strength of each product we were automating KF testing on

was run in triplicate using the manual (currently validated method) and

automated KF (potential new method supplement). Results were then analyzed and compared using a statistical analysis comparing 95% confidence of our data.

Product	Strength	Manual Replicates	Automated Replicates
Product A	High	3	3
	Middle	3	3
	Low	3	3
Product B	High	3	3
	Middle	3	3
	Low	3	3
Product C	High	3	3
	Middle	3	3
	Low	3	3

Design of Experiment: Robustness and Intermediate Precision

Robustness: altering spin speed of polytron, extraction time/stir time, covering of vessel using foil. *Products tend to be hygroscopic meaning they gain water over time.

Experiment	Polytron Spin Speed	Extraction Time	Vessel Coverage
1	Speed 1	Extraction Time 1	Coverage 1
2	Speed 1	Extraction Time 1	Coverage 2
3	Speed 1	Extraction Time 1	Coverage 3
4	Speed 1	Extraction Time 2	Coverage 1
5	Speed 1	Extraction Time 2	Coverage 2
6	Speed 1	Extraction Time 2	Coverage 3
7	Speed 1	Extraction Time 3	Coverage 1
8	Speed 1	Extraction Time 3	Coverage 2
9	Speed 1	Extraction Time 3	Coverage 3
10	Speed 2	Extraction Time 1	Coverage 1
11	Speed 2	Extraction Time 1	Coverage 2
12	Speed 2	Extraction Time 1	Coverage 3
13	Speed 2	Extraction Time 2	Coverage 1
14	Speed 2	Extraction Time 2	Coverage 2
15	Speed 2	Extraction Time 2	Coverage 3
16	Speed 2	Extraction Time 3	Coverage 1
17	Speed 2	Extraction Time 3	Coverage 2
18	Speed 2	Extraction Time 3	Coverage 3
19	Speed 3	Extraction Time 1	Coverage 1
20	Speed 3	Extraction Time 1	Coverage 2
21	Speed 3	Extraction Time 1	Coverage 3
22	Speed 3	Extraction Time 2	Coverage 1
23	Speed 3	Extraction Time 2	Coverage 2
24	Speed 3	Extraction Time 2	Coverage 3
25	Speed 3	Extraction Time 3	Coverage 1
26	Speed 3	Extraction Time 3	Coverage 2
27	Speed 3	Extraction Time 3	Coverage 3

Intermediate Precision: altering day (to account for day to day humidity changes) and analyst (to account for variation in technique).

Day	Analyst
1	1
1	2
1	3
2	1
2	2
2	3

Automation of Karl Fischer Titration for Animal Pharmaceutical Product Testing at Elanco Animal Health

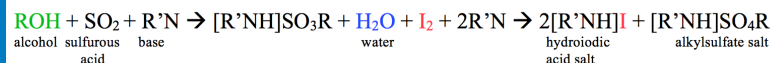
Elizabeth L. Grubbs

Karl Fischer Titration

Uses: Primary method of water content determination used worldwide by government, food science, academia, research, industry, and quality control.



Bunsen Reaction:



Manual Karl Fischer Instrument:

Sulfurous Acid and the Base in the Karl Fischer Reagent react with water molecules in the vessel. This creates an intermediate that goes on to react with the solvent (typically Methanol) and produces a product with no water molecules. This is theoretically 'dry'. Once the intermediate alkyl sulfite salt is produced, it is oxidized by iodine to the alkyl sulfate salt and this oxidation reaction consumes water. The reaction from I₂ to I⁻ is picked up by a conductivity probe and produces a titration curve as shown in order to find the titration endpoint. %Water of the sample is quantified using the mass of sample added and volume of titrant consumed. %Water (w/w) = (mL KF Reagent consumed x KF Titer x 100)/(g of sample x 100).

Project:

Automate the Karl Fischer titration use for testing of products at Clinton labs.

Benefits: Time and cost savings, ergonomic benefits, increased precision and accuracy.

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Results and Discussion:

My work was all accomplished successfully and new official documentation as well as validation protocol was written in order to implement this work for testing in the Quality Control labs for Elanco Clinton Manufacturing. I also created a training guide and hosted a training to teach analysts how to operate the new instrument.

Future Work

This process could be used for products beyond those that I did testing for. Additional experiments would need to be run. I have been hired as a chemist and will be returning next summer as a full-time employee!

Things I Learned Working in Industry

1. The work is fast-paced and everything you touch at a manufacturing site has to go out the door to customers despite problems that arise.
2. A lot of things are extremely company confidential due to competitors in the market.
3. The manufacturing site in Clinton is huge - the third largest fermentation site in the country right beneath two Anheuser-Busch sites. There are a lot of steps in the process and a lot of people that make the site run efficiently.
4. There is a lot of work regarding official documentation practices including lab notebooks that is very different from typical research.

Acknowledgements

1. Elanco Animal Health - specifically Elanco Clinton Laboratories
2. Patrick Cheek - Chemist, my technical mentor

References

1. Elanco Animal Health Confidential Documents and Data

